REMARKS

This patent application includes Claims 1-61 New claims have been added to restore some of the dependencies eliminated by deleting multiple dependencies.

Respectfully submitted,

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2598/1G196US1

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Sang Kyeong YUN and Dong Hoon KIM

Filed: CONCURRENTLY

For: PIEZOELECTRIC/ELECTROSTRICTIVE FILM ELEMENT FORMED AT LOW TEMPERATURE USING ELECTROPHORETIC DEPOSITION

AMENDMENT MARK-UP SHEET FOR PRELIMINARY AMENDMENT

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Sir:

In the Claims:

Please amend Claims 22 and 41 to read as follows:

22. (Amended) The piezoelectric/electrostrictive film element in [Claim 20 or] Claim 21, wherein the method further comprises a step of drying said piezoelectric/electrostrictive film between g) and h).

41. (Amended) The piezoelectric/electrostrictive film element in [Claim 39 or] Claim 40, wherein the method further comprises a step of drying the piezoelectric/electrostrictive film between g) and h).

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Docket No.: 2598/1G196US1

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Sang Kyeong YUN ET AL.

Filed: Concurrently

For: PIEZOELECTRIC/ELECTROSPTRICTIVE FILM ELEMENT AT LOW TEMPERATURE USING ELECTROPHORETIC DEPOSITION

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~~METHOD FOR FORMING~~ PIEZOELECTRIC/ELECTROSTRICTIVE FILM ELEMENT FORMED AT LOW TEMPERATURE USING ELECTROPHORETIC DEPOSITION ~~AND THE FILM ELEMENT FORMED BY THE METHOD~~

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates to a ~~method for forming~~ piezoelectric/electrostrictive film element formed by a method using an ultrafine ceramic oxide powder and the electrophoretic deposition, and piezoelectric/electrostrictive film element produced by the method. In particular, the present invention relates to a ~~method for forming~~ a piezoelectric/electrostrictive film element formed at low temperature by way of electrophoretic deposition ~~method~~ using an ultrafine ceramic oxide powder having excellent reactivity and produced by a single process at low temperature~~, and the piezoelectric/electrostrictive film element produced by the method~~.

Description of the Prior Art

Unit particle micronization and uniformity of particle diameter distribution ~~uniformalization etc~~. are emphasized in ceramic oxide powder which is a raw material of various devices using the ceramics such as ink jet head, memory chip, and piezoelectric substance, This is because in case of finer particles the activation energy can be lowered by surface treatment and the reactivity and applicability can be improved by particle electrification.

So far the method has been used where a ceramic sol with controlled ~~of~~ viscosity or a ceramic oxide powder regenerated by a suitable solvent is fixed at the substrate in order to form a

piezoelectric/electrostrictive film element in a manufacturing method of various film devices using the ceramics.

Considering the ultimately obtained film quality, methods mainly used for the ceramic sol solution are dip coating, spin coating, electrochemical oxidation/reduction etc. while methods used for the ceramic oxide powder are various printing, molding, electrophoretic deposition (EPD) etc.

Among these methods, EPD is a method to mold an elaborate film, ~~using~~ making use of the polarization of each component by electric polarity and the stacking property of solid particles.

In the EPD process using a ceramic oxide powder, shown in block diagram form in Figure 2, ceramic particles of average diameter not less than 1 μm made by a solid phase process are dispersed in an adequate dispersion medium of water or organic dispersant. Then, they are mixed with a pH-controlling medium to make a sol solution controlled ~~of~~ in surface electric charge, which the colloidal suspension is used for ceramic to move to a cathode or anode to form a film on a substrate. ~~which~~ This film is vapor deposited by thermal treatment above 1000°C, eventually to, form the film.

EPD like this has an advantage to make a high quality film unrestricted ~~of~~ in area or thickness, using a simple equipment.

But ~~there needs~~ a separate operation is needed to disperse powder using a dispersant, in order to secure dispersibility, because a large particle diameter powder is used. ~~and~~ Also, there is inevitability a problem of high temperature thermal treatment to get material property peculiar ~~of~~ to the ceramic, because the formed film property is similar to the bulk.

SUMMARY OF THE INVENTION

The present invention, to solve the problems, has a purpose of providing ~~firstly~~ a ~~method to form~~ a piezoelectric/electrostrictive film element formed by ~~through~~ electrophoretic deposition and thermal treatment at low temperature using ultrafine ceramic oxide powder, which is ~~very~~ excellent in reactivity and has ~~as well as it is~~ very fine ~~in~~ particle size, as it has been made by a single process at low temperature by a combustion method using ~~the~~ citric acid as a combustion aid ~~and of secondly the provision and supply of~~ piezoelectric/electrostrictive film element formed by the method ~~at low temperature~~.

The present invention to achieve the purpose, provides ~~features~~ a ~~method for forming~~ a piezoelectric/electrostrictive film element former at low temperature using electrophoretic deposition, by the method comprising the steps of : preparing a solution or a dispersed mixture containing constituent ceramic elements by dissolving or dispersing the raw material of constituent ceramic elements in a solvent or a dispersion medium; preparing a mixed solution by adding citric acid into the solution or the dispersed mixture in which the constituent ceramic elements are dissolved or dispersed; getting ultrafine ceramic oxide powder of particle size less than 1 μm with uniform particle diameter size distribution, by forming ceramic oxide without scattering over, by a nonexplosive oxidative-reductive combustion reaction by thermally treating the mixed solution at 100-500°C; preparing a suspension by dispersing the ultrafine ceramic oxide powder in an organic dispersant; preparing a ceramic sol solution by dissolving constituent ceramic elements of the same or similar constituent ~~with~~ as the ultrafine ceramic oxide powder in water or

an organic solvent; dispersing by mixing with the ceramic sol solution the suspension in which the ultrafine ceramic oxide powder is dispersed ~~with the ceramic sol solution~~; forming a piezoelectric/electrostrictive film element by submerging a substrate into the suspension in which the ultrafine ceramic oxide powder and the ceramic sol solution are mixed and then ~~by~~ performing electrophoretic deposition; and thermally treating the piezoelectric/electrostrictive film element at 100-600°C , so that the solvent is removed by the thermal treatment and ~~the~~ bonding among the ultrafine ceramic oxide powder particles is induced, while the ceramic sol acts as a reaction medium on the surfaces of the ceramic oxide particles.

Also the present invention features a piezoelectric/electrostrictive film element produced by a method comprising the steps of : preparing a solution or a dispersed mixture containing constituent ceramic elements by dissolving or dispersing the raw material of constituent ceramic elements in a solvent or dispersion medium; preparing a mixed solution by adding citric acid into the solution or the dispersed mixture in which the constituent ceramic elements are dissolved or dispersed; getting ultrafine ceramic oxide powder of particle size less than 1 μm with uniform particle diameter size distribution by forming ceramic oxide without scattering over, by a nonexplosive oxidative-reductive combustion reaction by thermally treating the mixed solution at 100-500°C ; preparing a suspension by dispersing the ultrafine ceramic oxide powder in an organic dispersant; preparing a ceramic sol solution by dissolving constituent ceramic elements of the same or similar

constituent ~~with~~ as the ultrafine ceramic oxide powder in water or an organic solvent; dispersing by mixing with the ceramic sol solution the suspension in which the ultrafine ceramic oxide powder is dispersed ~~with the ceramic sol solution;~~ forming a piezoelectric/electrostrictive film element by submerging a substrate into the suspension in which the ultrafine ceramic oxide powder and the ceramic sol solution are mixed and then ~~by~~ performing electrophoretic deposition; and thermally treating the piezoelectric/electrostrictive film element at 100-600°C, so that the solvent is removed by the thermal treatment and ~~the~~ bonding among the ultrafine ceramic oxide powder particles is induced, while the ceramic sol acts as a reaction medium on the surfaces of the ceramic oxide particles.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a flow diagram ~~producing~~ for method ~~of~~ for producing ultrafine ceramic oxide powder used in the present invention.

Figure 2 is a flow diagram of ~~forming~~ a process ~~of~~ for forming piezoelectric/electrostrictive film element using the conventional electrophoretic deposition.

Figure 3 is a flow diagram of a method for forming a piezoelectric/electrostrictive film element using ~~the~~ electrophoretic deposition at low temperature according to the present invention.

DETAIL DESCRIPTION

The present invention will be explained in detail.

First, a method for producing an ultrafine ceramic oxide powder used as a raw material in a piezoelectric/electrostrictive film element produced according to the present invention as in

~~a~~ The flow diagram of Figure 1 will be explained.

A n ultrafine ceramic oxide powder ~~producing~~ manufacturing method of the present invention comprises the steps of: sufficiently dissolving or uniformly dispersing the raw material of constituent ceramic elements in a solvent or dispersant to make a solution or a dispersion mixture containing the constituent ceramic elements; adding, into the solution or the dispersion mixture containing the constituent ceramic elements, citric acid in no less than the required amount to give rise to an oxidative-reductive combustion reaction with an anion of the ceramic constituent ceramic element so as to make a mixed solution; and thermally treating the mixed liquid at 100-500°C. But it may additionally further comprises a step of conducting additional thermal treatment at 700-900°C to increase crystallinity.

As for the raw material containing the constituent ceramic elements, use is made of ~~from among~~ oxide, carbonate, nitrate etc. of constituent ceramic element, its salt with organics or inorganics, or a constituent ceramic element~~s~~ complex.

As for the constituent ceramic element, it is preferable to use a piezoelectric/electrostrictive ceramic element comprising lead (Pb) and titanium (Ti) as basic constituent elements.

Especially as ~~for~~ to the constituent ceramic element, it is preferable to use that composed of elements including lead (Pb), zirconium (Zr) and titanium (Ti), or lead (Pb), zirconium (Zr), titanium (Ti) / lead (Pb), magnesium (Mg), niobium (Nb).

As for the solvent, or the dispersant to dissolve or disperse the raw material of the constituent ceramic elements, one

or more are selected ~~to use~~ from among water and organic solvents that can dissolve or disperse the raw material containing the constituent ceramic elements. As for the organic solvents, mainly acetic acid, dimethyl formamide, methoxyethanol, alcohols, or glycols ~~etc.~~ are used.

As for the combustion aid, citric acid is used, which is an organic compound that can give rise to a combustion reaction. In the conventional method, ~~the~~ citric acid has been used not as a combustion aid but as a complexing agent in order to give reaction uniformity, and it has been used in processes such as the Pechini process, where a speed-controlled combustion reaction can be induced using citric acid's flammability and complex formation effect.

A mixture is made by adding citric acid into a solution or a dispersed mixture where constituent ceramic elements are dissolved or dispersed. The quantity of the citric acid added shall not be less than the necessary amount to give rise to an oxidative-reductive combustion reaction with the anion of the constituent ceramic element. Reaction speed can be controlled by the quantity of citric acid added.

The mixture made by the addition of the citric acid is thermally treated at 100-500°C. Though the crystallinity of the ceramic phase increases ~~as~~ with the temperature for the thermal treatment, the citric acid combustion reaction may start enough if ~~only~~ the temperature for the thermal treatment is over 100°C. ~~And~~ Although the reaction can arise even if the temperature for the thermal treatment is above 500°C, thermally treating above that temperature is meaningless when compar~~ing~~ed with the conventional method.

More preferably, it shall be thermally treated at 150-300°C, which is a temperature range that can secure suitably the crystallinity of the ceramic phase, although it is a considerably low temperature range for a thermal treatment.

If the mixture is thermally treated to vaporize the solvent or the dispersant, the added citric acid acts as a reductive combustion aid and is removed, giving rise to a nonexplosive oxidative-reductive combustion reaction with the anion of a constituent ceramic element, when the ceramic oxide is formed without scattering out by virtue of reaction heat generated at this time.

And in the reaction, components other than the constituent ceramic element are removed ~~during~~ after a sufficient time of combustion reaction so that the ultrafine ceramic oxide powder of pure type, without impurity is obtained.

The particle size of the ultrafine ceramic oxide powder obtained by the method is below 1 μm, and is specifically 0.01-0.1 μm so extremely fine with uniform powder particle diameter distribution. The primary particles of ~~which~~ the powder exist as independent bodies or as a soft aggregate type, and are in completely burnt ceramic phase so that the weight does not decrease even by additional thermal treatment.

~~And~~ Because the powder has excellent surface reactivity, so that molding is feasible even only with a thermal treatment at low temperature, the degree of freedom for a vibration plate is high and diverse methods of printing and coating can be applied.

But it may additionally comprise~~s~~ a step of conducting additional thermal treatment of the obtained ultrafine ceramic

oxide powder at 700-900°C to increase the crystallinity of the powder produced.

A method for forming a piezoelectric/electrostrictive film element at low temperature by an electrophoretic deposition process using ultrafine ceramic oxide powder will be explained. Figure 3 ~~shows~~ represents a method for forming a piezoelectric/electrostrictive film element at low temperature by an electrophoretic deposition process.

As for the ceramic oxide powder, the ultrafine ceramic oxide powder obtained by the method is used because it is effective to use fine powder to secure a system feasible of forming at low temperature, considering the powder reactivity itself.

The ultrafine ceramic oxide powder produced has small ceramic particle size with uniform size distribution and no voids, so that it can ~~get~~ achieve ideal stacking result with maximum bonding strength between particles.

~~Whence~~ It is preferable to use PZT, PMN or their solid solution (PZT-PMN) complex oxides ~~as~~ for the ultrafine ceramic oxide powder.

~~And~~ The ultrafine ceramic oxide powder may additionally comprise~~s~~ one or more components among nickel (Ni), lanthanum (La), barium (Ba), zinc (Zn), lithium (Li), cobalt (Co), cadmium (Cd), cerium (Ce), chromium (Cr), antimony (Sb), iron (Fe), yttrium (Y), tantalum (Ta), tungsten (W), strontium (Sr), calcium (Ca), bismuth (Bi), tin (Sn) and manganese (Mn).

Because interparticle vacancy exists no matter how closely it approaches an ideal stacking state, in order to improve the density

problem occurring according to the interparticle vacancy, there are separately prepared: a suspension or a dispersion liquid ~~dispersed of~~ comprising the ultrafine ceramic oxide powder in an organic dispersant, and a ceramic sol solution having the same or similar composition with the ultrafine ceramic oxide powder.

The ultrafine ceramic oxide powder which is used is dispersed in an organic dispersant, ~~as~~ for which ~~is~~ are mainly used alcohols such as ethanol and methoxy ethanol, and acetones such as acetone and acetyl acetone.

It is preferable that the content of the organic dispersant is 1-500 ml per gram of the ultrafine ceramic oxide powder. ~~It~~ This is used because adequate dispersion does not arise if the content of the organic dispersant is lower than 1 ml per gram of the ultrafine ceramic oxide powder, while if the content is higher than 500 ml per gram of the ultrafine ceramic oxide powder, ~~then~~ the oxide powder is diluted to be of an exceedingly low viscosity.

The ceramic sol solution is made based on water or organic solvent which can be used from among a variety of organic solvents but is preferable to be mainly acetic acid, dimethyl formamide, methoxyethanol, alcohols, glycols ~~etc~~.

Then the ceramic sol solution and the suspension of the ultrafine ceramic oxide powder which are prepared separately are mixed. ~~The~~ A preferred mixing ratio of the ultrafine ceramic oxide powder and the ceramic sol solution ~~may be preferable if~~ is that the ~~content of the~~ ceramic sol solution is 1-500 parts by weight based on the weight of the ultrafine ceramic oxide powder when the powder and the suspension are mixed.

Thus if the ultrafine ceramic oxide powder and the ceramic

sol solution are mixed, ~~the~~ most of the voids occurred after stacking is filled by the ceramic sol and the sol is transferred to the ceramic particles during the thermal treatment process, after the film formation, so that voids substantially decrease.

~~And~~ As the ceramic sol itself has electric charge and is compatible with both the ultrafine ceramic oxide powder and the solvent, it is feasible ~~of~~ to achieve stabilization of the suspension and the surface electricity charge treatment of the ultrafine ceramic oxide powder, even without a separate operation and pH control medium.

If a work electrode is attached ~~of~~ to the substrate and an opposite electrode dipped into the sol solution ~~mixed~~ made up of the ultrafine ceramic oxide powder and the ceramic sol solution, the ceramic sol and the ultrafine ceramic oxide powder polarized in the sol solution phase move to the work electrode to form a film on the substrate ~~attached at the work electrode~~.

Metal, resinous polymeric organic compound, or ceramics may be used as a vibration plate.

~~As~~ For the metal for the vibrating plate, nickel (Ni) or stainless steel is mainly used; as for the resinous polymeric organic compound, polyester, polyimide, or teflon resin is mainly used; and as for the ceramics, alumina (Al_2O_3), zirconia (ZrO_2), silicon (Si), silicon carbide (SiC), silicon nitride (Si_3N_4), silicon dioxide (SiO_2), or glasses is mainly used.

At this time, it may be postfinished after generally forming the film on the substrate, or a screen, mold, or mask might be set on the substrate so as to form a piezoelectric/electrostrictive film element of desired type.

~~Whence~~ It is preferable to form the piezoelectric/electrostrictive film element with a ~~in the~~ thickness of 1-100 μm, and ~~may be~~ more preferable to form it with a ~~in the~~ thickness of 5-30 μm.

The formed piezoelectric/electrostrictive film is thermally treated to remove the remaining solvent and to convert the contained sol into fine ceramic particles. Thus the solvent is removed by thermal treatment and the ceramic sol acts as a reaction medium on oxide particle surface to induce the bonding between ultrafine ceramic oxide particles.

The reason for the reaction, ~~is~~ sufficient only by the thermal treatment at ~~the~~ low temperature of 100-600°C, is that a the reaction same as a sintering may take place by mutual reaction of bonding between the ultrafine ceramic oxide powder and the raw material of the constituent ceramic elements in the ceramic sol solution. ~~And~~ So, the added organic materials are removed during the thermal treatment.

Specifically in case of the polymeric organic compound, because the substrate may be damaged if thermally treated above 500°C, it is preferable to thermally treat it at 100-300°C, ~~in case~~ where the polymeric organic compound is used as a substrate.

~~So~~ More preferably, thermal treatment may be conducted at 150-300°C, which temperature range can suitably secure the crystallinity and formability of the piezoelectric/electrostrictive film element, although ~~even as~~ the range is for the thermal treatment at considerably low temperature.

~~And~~ The method mat further comprises a step of drying the formed piezoelectric/electrostrictive film element before the

thermal treatment, after forming the piezoelectric/electrostrictive film element, ~~whence~~ and it is preferable to dry the piezoelectric/electrostrictive film element at 70-100°C.

The piezoelectric/electrostrictive film element obtained by the method is excellent in characteristics proper ~~of~~ ceramics, although the element has been thermally treated at low temperature.

~~So as~~ For the present invention as above, energy required for electrophoretic deposition ~~process~~ is reduced, because the ultrafine ceramic oxide powder is used and there is a low energization effect of the producing method, because the piezoelectric/electrostrictive film element can be formed ~~where~~ the stacking status of the particles is very dense even only with the thermal treatment at low temperature.

Now the present invention will be explained in detail by the following practical examples. But the following application examples are only illustrations of the present invention and do not ~~confine~~ limit the extent of the present invention.

[Example 1]

1 g of fine powder PZT-PMN was added into methoxyethanol 300 ml and acetyl acetone 20 ml, and 1 g of PZT sol was added into the mixed solution,. Then, it was dispersed for 30 minutes by an ultrasonic generator. Afterwards it was agitated by a magnetic stirrer.

A SUS 316L plate fixed ~~of~~ to the silicon substrate and mask was prepared as a work electrode and a SUS plate of the same area was prepared as an opposite charge electrode. The electrodes were put into the suspension and were connected to an electric supply to

pro~~ceed~~duce electrophoretic deposition at 70 V and 0.03 A for 10 minutes.

The work electrode ~~completed of~~ treated by vapor deposition was withdrawn, the substrate was separated from the SUS plate, and the mask was removed.

The substrate portion where a pattern had been formed was thermally treated at 100°C in a chamber and was dried, ~~which~~ and was then thermally treated at 300°C for 2 hr. Then aluminum was vapor deposited as an upper electrode, and electric potential was ~~added~~ applied to measure the displacement of the substrate (vibration plate) by piezoelectric phenomenon.

The piezoelectric characteristics represented by the displacement of the vibration plate ~~was more~~ were excellent and better than tha~~t~~ose of a piezoelectric/electrostrictive film element produced by the conventional method.

[Example 2]

1 g of fine powder PZT-PMN was added into methoxyethanol 300 ml and acetyl acetone 100 ml, ~~and~~ into which mixed solution, 4 g of PZT sol was added. Then it was dispersed for 30 minutes by a untrasonic generator. Afterwards it was agitated by a magnetic stirrer.

A SUS 316L plate fixed ~~of~~ to a nickel substrate and a mask was prepared as a work electrode and a SUS plate of the same area was prepared as an opposite charge electrode. Then the electrodes were put into the suspension and were connected to an electric supply to pro~~ceed~~duce electrophoretic deposition at 70 V and 0.03 A for 10 minutes.

The work electrode ~~completed of~~ treated by vapor deposition was

withdrawn, the substrate was separated from the SUS plate, and the mask was removed.

~~A~~ The substrate portion where a pattern had been formed was thermally treated at 70°C in a chamber and was dried, ~~which~~ and was then thermally treated at 300°C for 2 hr. Then gold was vapor deposited as an upper electrode, and electric potential was ~~added~~ applied to measure the displacement of the substrate (vibration plate) by piezoelectric phenomenon.

Piezoelectric characteristics represented by the displacement of the vibration plate ~~was more~~ were excellent and better than ~~that~~ those of a piezoelectric/electrostrictive film element produced by the conventional method.

ABSTRACT OF THE DISCLOSURE

~~The present invention relates to~~ A ~~method for forming~~ piezoelectric/electrostrictive film element formed at low temperature using electrophoretic deposition ~~, the method comprising~~ by method which includes the steps of: ~~preparing a solution or a dispersed mixture containing constituent ceramic elements by~~ dissolving or dispersing the raw material of constituent ceramic elements in a solvent or a dispersion medium; ~~preparing a mixed solution by~~ adding citric acid into the solution or the dispersed mixture ~~in which the constituent ceramic elements are dissolved or dispersed~~; ~~getting~~ obt. ultrafine ceramic oxide powder of particle size less than 1 μm with uniform particle diameter size distribution by forming ceramic oxide ~~without scattering over,~~ by a nonexplosive oxidative-reductive combustion reaction by thermally treating the mixed solution at 100-500°C; preparing a suspension by dispersing the ultrafine ceramic oxide powder in an organic dispersant; preparing ceramic sol solution by dissolving constituent ceramic elements of the same or similar constituent ~~with~~ as the ultrafine ceramic oxide powder in water or an organic solvent; ~~dispersing by~~ mixing the suspension ~~in which the ultrafine ceramic oxide powder is dispersed~~ with the ceramic sol solution; forming a piezoelectric/electrostrictive film element by submerging a substrate into ~~the suspension which the ultrafine ceramic oxide powder and the ceramic sol solution are mixed~~ the mixture and then by performing electrophoretic deposition; and thermally treating the piezoelectric/electrostrictive film element at 100-600°C.

~~Also the present invention relates to a piezoelectric/electrostrictive film element produced by the~~

method. Whose advantageous effects are that energy required for electrophoretic deposition process is reduced and piezoelectric/electrostrictive film element can be formed where stacking status of particles is very dense even only with low temperature treatment.